

Rapid Sediment Characterization (RSC) Technologies

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Outline

- Navy Need
- Sediment Screening Technologies
 - X-Ray Fluorescence (XRF)
 - Ultraviolet Fluorescence (UVF)
 - Immunoassay for Organics (IAO)
 - QuikSed Biological Screen (QwikSed)
- Regulatory Issues
- Case Studies
- Role in the Ecological Risk Assessment Process
- Summary and Conclusions
- References
- Information Sources and Tech Transfer Tools

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Problem

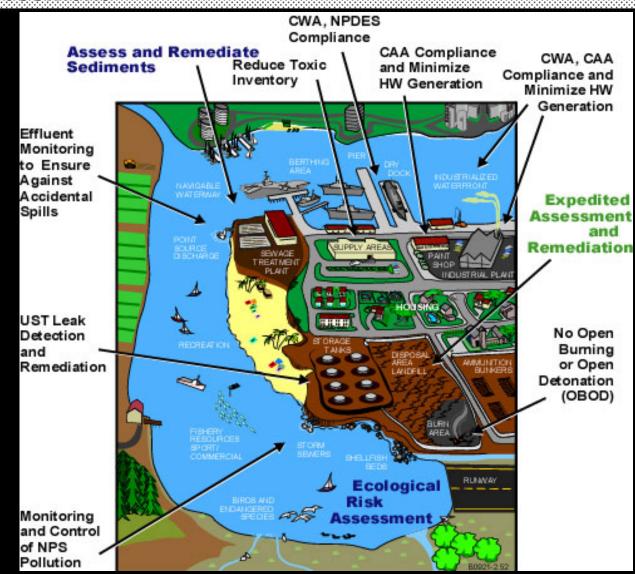
Managing Contaminants in the Marine Sediment Environment

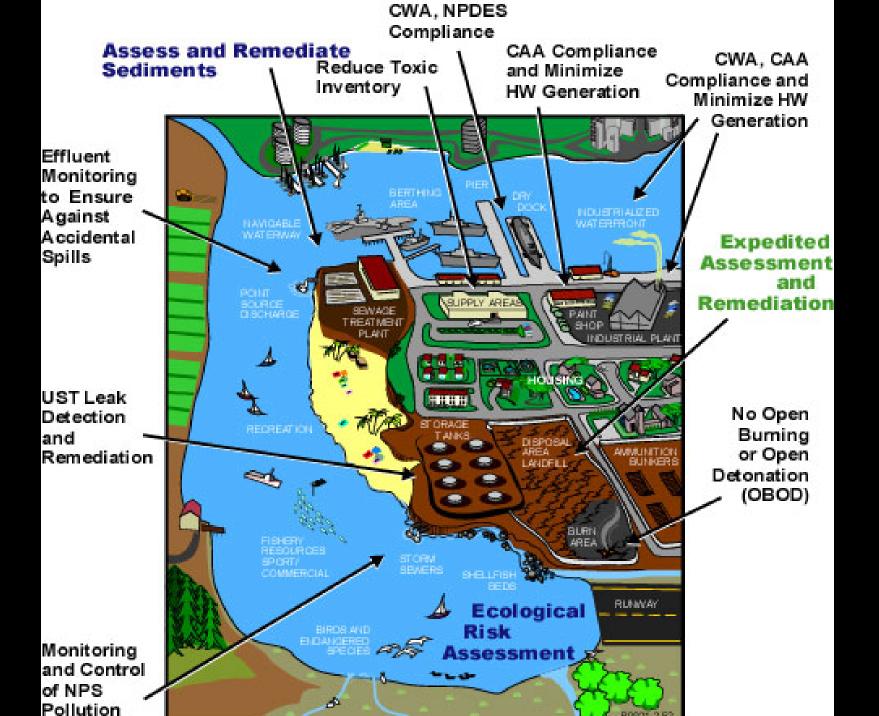
- Where are the contaminants?
 - Historical documents, site assessment, RSC
- What are the contaminants?
 - Laboratory analytics
- How do we determine if contaminants are a problem?
 - Standard U. S. EPA bioassay tests, risk assessment tools
- What do we do about contamination?
 - Management, monitoring, remediation
- How do we prevent contamination in the future?

Problem

Marine Sediment Assessment

- Marine sediments
 are the ultimate
 receptor of effluent
 from all Navy
 activities, both at
 sea and on shore
- As a result, regulation of sediments can impact all Navy activities





Today's Situation

- As regulatory attention moves to sediment systems, many aspects of sediments will be under scrutiny
- Navy has many sediment sites which are already in the Remedial Investigation/Feasibility Study (RI/FS) process
- RSC tools fit well into the eight-step eco-risk process that the Navy is implementing at these sites
- By extrapolation of Superfund work, current U.S. EPA policy leads to removal of impacted sediments as a presumptive remedy
- Given volumes potentially involved, cost will rapidly run to the \$billions

Navy Need

- A growing body of evidence suggests that sediment removal can at times result in more ecological damage, rather than improvement
- The Navy must assess and manage contaminated sediments to conduct dredging, base closures, or to clean up contaminated "hot spots"
- For contaminated sediments, optimum procedures have not been determined to:
 - Adequately assess extent of contamination, potential toxicity, and environmental effects
 - Manage those factors deemed to be a risk

Navy Need

- While the Navy and many other groups have expended considerable resources developing "innovative" sediment technologies, these technologies are not getting to the users in a useful way (incomplete tech transfer)
- Single tools rarely stand alone for complex environmental issues, and there has not been a critical analysis of how these approaches replace or enhance standard procedures

Navy Need

RSC Technologies

- 1.III.1.k (High)* Pillar: Cleanup
 - Improved Field Analytical Sensors, Toxicity Assay Methods, and Protocols to Supplement Traditional Sampling and Lab Analysis
- 2.II.2.b (High)* Pillar: Compliance
 - Improved Field Analytical Sensors, Toxicity Assay Methods, and Protocols to Supplement Traditional Sampling and Lab Analysis

*DON Requirements are from "Environmental Quality RDT&E Strategic Plan"

Regulatory Driver

Environmental Risk Assessment

 Navy policy* specifically requires that sampling programs focus primarily on the identification of potential contaminant sources and on the delineation of areas of contaminated media. It further dictates that sampling programs should make use of advanced chemical and biological screening technologies, data quality objectives, and statistical procedures to minimize overall sampling requirements.

*Chief of Naval Operations (CNO) Letter 5090 Ser N453E/9U595355 dated 05 April 1999; Navy Policy for Conducting Ecological Risk Assessments.

Part of the Solution...

What are RSC Tools?

- Field transportable analytical tools which provide measurements of chemical, biological or physical parameters on a real-time or near realtime basis
- Often commercial off-the-shelf (COTS) units
- Tools can be used individually or in concert depending on data needs
- Examples of tools
 - Chemical Measurements
 - X-Ray Fluorescence (XRF) for Metals
 - Ultraviolet Fluorescence (UVF) for PAHs
 - Immunoassay for Organics (PCBs, PAHs, and Pesticides)
 - Biological Measurements
 - QwikSed Bioassay for Biological Effects
 - Physical Measurements
 - Particle Size, Moisture, Density

Why use RSC Tools?

- Utilize RSC along with standard lab data to:
 - Reduce number of costly laboratory analyses
 - Map contaminated sediment volumes more efficiently (at less than 50% of current costs) to reduce remediation costs
 - Increase the probability of successful, high-impact sampling
 - Provide the ability to fill in gaps and reduce uncertainty at several steps of the RI/FS process without the enormous cost of traditional resampling efforts

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- Applicability
- Principles of Operation
- Cost
- Advantages and Limitations

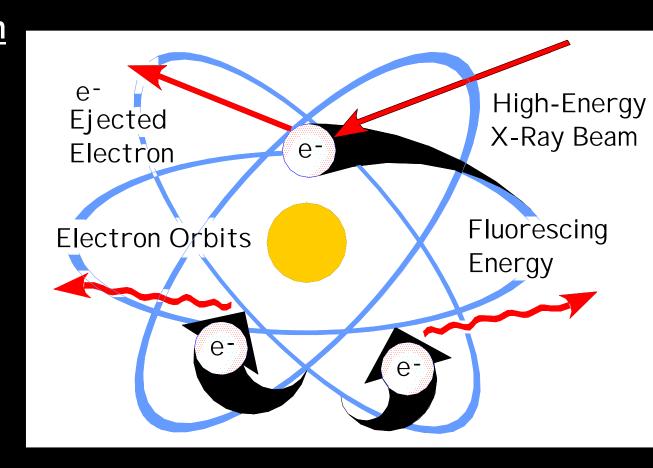
XRF for Metals



XRF for Metals

Principle of Operation

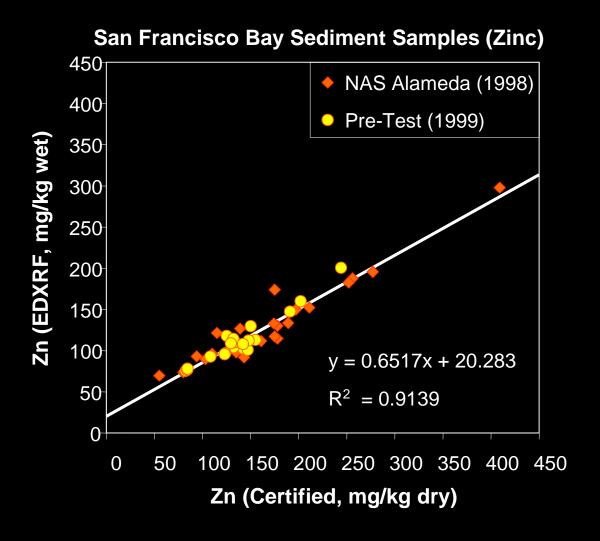
Samples are exposed to x-ray energy, which results in x-ray fluorescence (XRF). The type (energy level) of fluorescence identifies which metals are present and its intensity is proportional to concentration.



XRF Lab Validation

XRF for Metals

- Metals data show high correlation coefficients (r²) when compared to lab data (r² from 0.7 to 0.9)
- Data are not site specific, similar results obtained at multiple sites



Cost Per Sample

XRF for Metals

	Analysis Method	RSC Tool* (\$25/sample)	Laboratory (\$300/sample)	Total Cost
T	RSC: X-ray Fluorescence	400 samples	100 samples (25% validation)	\$40K
A L S	Certified Lab: ICP/MS	0 samples	400 samples	\$120K

^{*}Capital investment not included; cost per analysis only

Advantages and Limitations

XRF for Metals

<u>Advantages</u>

- Minimal sample preparation
- Multi-element analysis
- Near real-time analysis
 - 2 to 5 min
- Low-cost analysis
- Detection limits*
 - Cu (20 to 100 ppm)
 - Zn (20 to 100 ppm)
 - Pb (10 to 50 ppm)

Limitations

- Semi-quantitative
- Matrix interferences
- Non-specific (e.g., cannot differentiate Cr⁺³ vs. Cr⁺⁶)
- Not suitable for all metals (e.g. Hg)

^{*}Low end detection limits are for bench-top EDXRF

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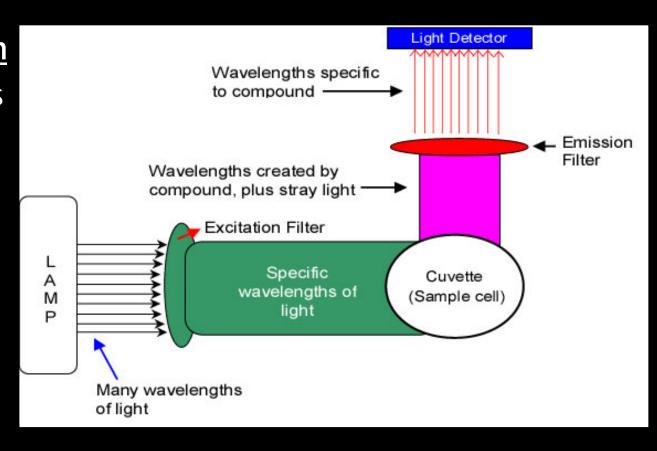
UVF for PAHs



UVF for PAHs

Principle of Operation

When ultraviolet light is passed through a sample extract, the sample emits light (fluorescence) proportional to the concentration of the fluorescent molecules (PAHs) in the sample

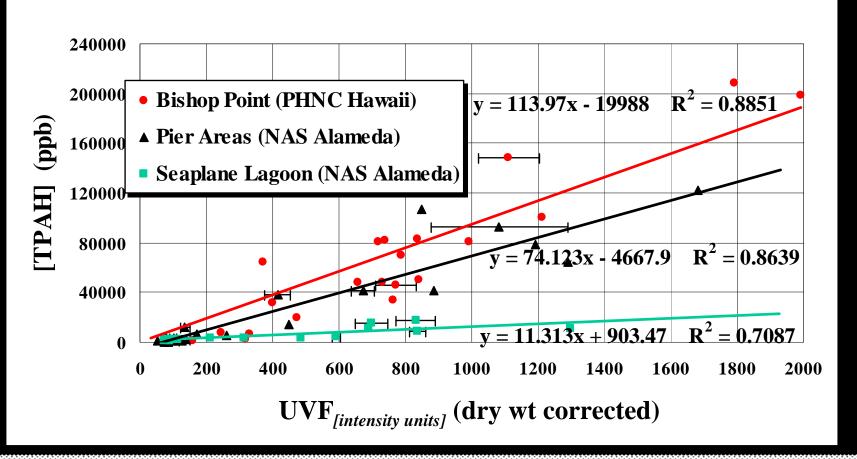


UVF Lab Validation

UVF for Total PAHs

Data are site specific, depending on PAH mixture (r2 from 0.7 to 0.9)

Ultraviolet Fluorescence (UVF) as Total PAHs



Cost Per Sample

UVF for Total PAHs

	Analysis Method	RSC Tool* (\$50/sample)	Laboratory (\$400/sample)	Total Cost
P A	RSC: UV Fluorescence	400 samples	100 samples (25% validation)	\$60K
Н	Certified Lab: GC/MS	0 samples	400 samples	\$160K

^{*}Capital investment not included; cost per analysis only

Advantages and Limitations

UVF for Total PAHs

<u>Advantages</u>

- Minimal sample preparation
- High throughput
 - 20 samples per day
- Near real-time analysis
 - 10 to 30 min
- Detection limit:
 - 1 to 5 ppm tPAH

Limitations

- Semi-quantitative
- Matrix sensitive
- Non-specific (cannot speciate different PAHs)
- Site-specific calibration required

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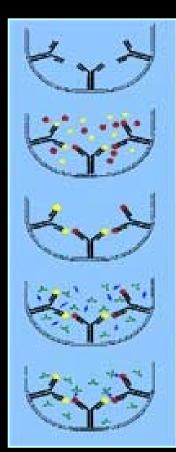
Immunoassay for Organics



Immunoassay for Organics

Principle of Operation

Antibodies are developed specifically to bind with organic compounds (e.g. PCBs, PAHs, pesticides), and that selective response is used to confirm the presence of the contaminant in samples. Color change in an extract solution is related to chemical concentration, with a spectrophotometer used to quantify the concentration.



Start with an antibody-coated tube or well.

Add sample and labeled antigen. Labeled and unlabeled antigens compete for a limited number of binding sites.

Remove unbound antigen.

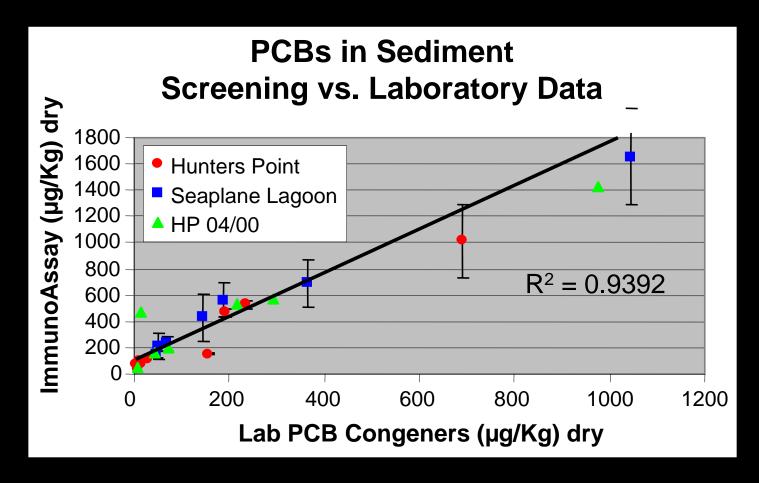
Add substrate and chromogen. Antibody
Antigen in sample
(analyte)
Labeled antigen
Substrate
Chromogen

Enzyme-substrate reaction causes chromogen to turn color. Less color means more analyte.

Immunoassay Lab Validation (PCBs)

Immunoassay for Organics

Immunoassay data for total PCBs show good correlation to lab data ($r^2 = 0.9$)



Cost Per Sample

Immunoassay for Organics

P C B

Analysis Method	RSC Tool* (\$25/sample)	Laboratory (\$400/sample)	Total Cost
RSC: Immunoassay	400 samples	100 samples (25% validation)	\$50K
Certified Lab: GC/ECD	0 samples	400 samples	\$160K

^{*}Capital investment not included; cost per analysis only

Advantages and Limitations

Immunoassay for Organics

Advantages

- High throughput/data density
- Rapid turnaround
 - 50 samples in a day
- Solvent extract can be used for PAH analysis
- Detection limit:
 - 50 to 500 ppb depending on dilution series

Limitations

- Matrix sensitive
- Cannot speciate between different Aroclor mixtures or individual congeners
- Tests require stringent attention to protocol

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QuikSed Biological Screen (QwikSed)

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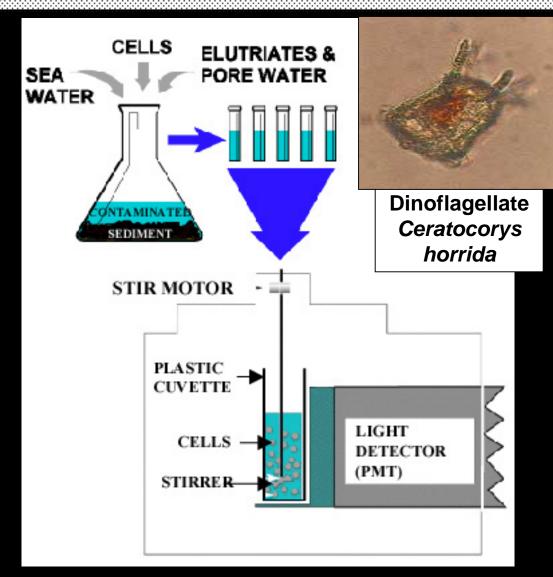
QwikSed Biological Screen



QwikSed Biological Screen

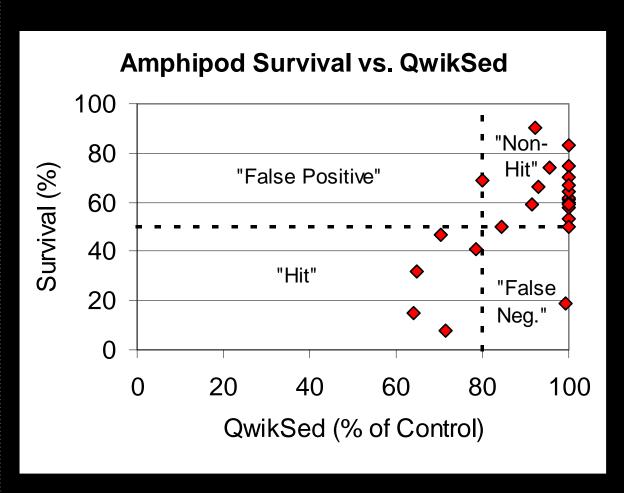
Principle of Operation

The QwikSed Bioassay measures the inhibition of light emitted by marine bioluminescent dinoflagellates (e.g., Ceratocorys horrida) exposed to a test solution (effluents, elutriates, or sediment pore waters). Any decrease in light output relative to controls suggests bioavailable contaminants or other stressors.



Lab Validation

QwikSed Biological Screen



- Good relationship to laboratory bioassay
 - 20% Hits
 - 72% Non-Hits
 - 4% False Positives
 - 4% False Negatives
- Various lab bioassays show as much or more variability to each other than to RSC data

Cost Per Sample

QwikSed Biological Screen

	Analysis Method	RSC Tool* (\$200/sample)	Laboratory (\$1200/sample)	Total Cost
TOXICITY	RSC: QwikSed Bioassay	400 samples	100 samples (25% validation)	\$200K
	Certified Lab: Standard Amphipod 10- day Bioassay	0 samples	400 samples	\$480K

^{*}Capital investment not included; cost per analysis only

Advantages and Limitations

QwikSed Biological Screen

<u>Advantages</u>

- Less time-consuming than standard bioassays
- 24- to 48-hour turnaround
- Sensitivity equivalent to other standard bioassays

Limitations

- Sensitive to confounding factors (e.g., ammonia)
- Non-specific
 - Results do not indicate of class of contaminant causing toxicity

Comparison of Methods

RSC vs. Laboratory Analyses

RSC (Screening)	Laboratory Analyses
AdvantagesRapid results can guide sampling locationsPotential for high data density for mapping	AdvantagesStandard methods that are very quantitativeCan often remove interferences
LimitationsOften non-specificSemi-quantitativeMatrix sensitive	Limitations • Often blind sampling • Long delays to results • Expensive (\$K/sample)
Cost per Sample • XRF (metals): \$25 • UVF (PAHs): \$50 • Immunoassay (PCBs): \$25 • QwikSed: \$200	Cost per Sample ICP/MS (metals): \$300 GC/MS (PAHs): \$400 GC/ECD (PCBs): \$400 Amphipod bioassay: \$1200
 Throughput XRF: 40 samples per day UVF: 20 samples per day Immunoassay: 50 samples per day QwikSed: 6 to 12 samples per day 	Throughput • Metals, PAHs, Bioassay: 30 to 90 days for data turnaround is not unusual

Benefits/Payback

RSC vs. Laboratory Analyses

- Laboratory analyses
 - Standard lab costs ~ \$1,000 chem + ~ \$1,000 bio
 - 400 samples cost \$800k
- Screening + laboratory analyses
 - Screen all 400, send 100 to the lab
 - \$300/sample to screen = \$120k
 - Screening of 400 + laboratory cost for 100 = \$320k

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Regulatory Issues

RSC Technologies

- RSC techniques have gained regulatory acceptance (see next slide and references at end of presentation)
 - Fit within U.S. EPA/USACE Dredge guidelines
 - Fit within U.S. EPA 8-step EcoRisk guidelines
- RSC techniques have been demonstrated/validated under several U.S. EPA and DoD programs
 - EPA ETV, SITE Program
 - DoD ESTCP Program

Regulatory Issues

RSC Technologies

 "It has become a widespread misconception that EPA "approves" (in a restrictive sense) which methods may be used to generate data within the RCRA or Superfund programs, and that these methods must be used as written in SW-846. The reasoning then becomes that new technologies or analytical methods cannot be used unless they appear in SW-846. This is a myth! An August 7, 1998 Memorandum reiterates previous EPA guidance that "SW-846" methods need not be applied in a prescriptive manner." Additional discussion about the relationship between SW-846, the Performance Based Measurement System (PBMS) and the use of innovative analytical technologies is provided in a accompanying summary. More information about PBMS can be found on the OSW PBMS webpage."*

*Quote from website address http://clu-in.org/char1.htm

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RSC Technologies

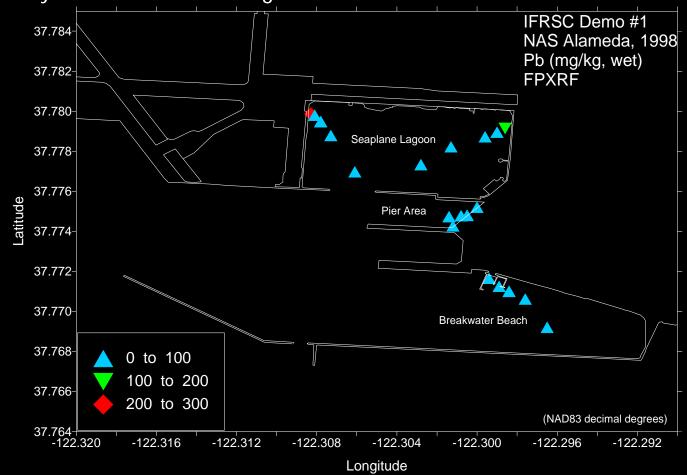
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NAS Alameda, CA: XRF

FPXRF Screening: Lead

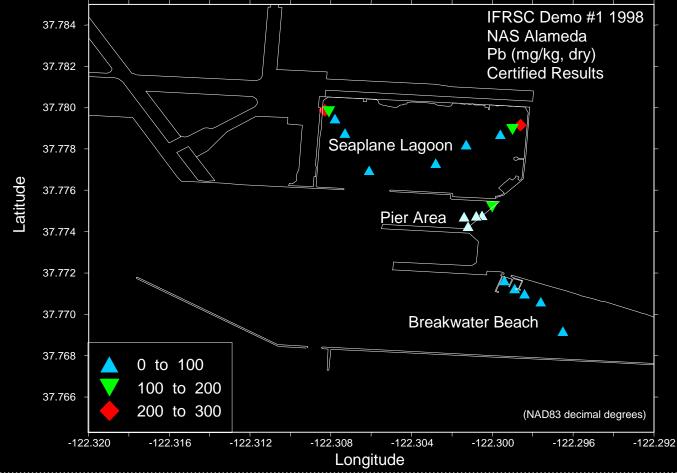
Lead primarily found in corners of lagoon



NAS Alameda, CA: XRF

Certified Results: Lead

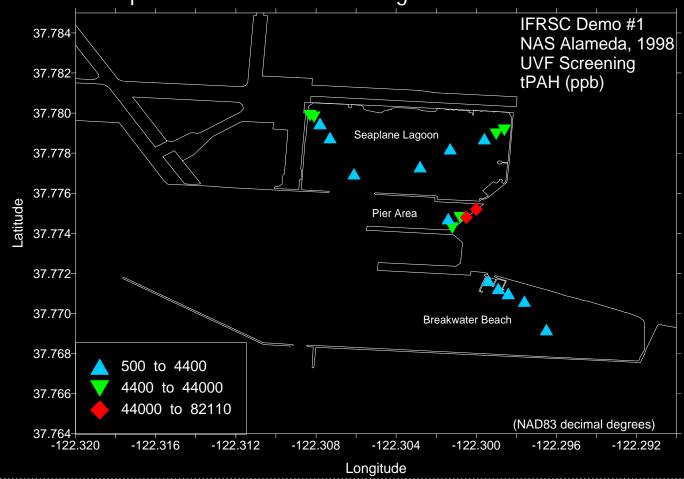
Laboratory data show patterns similar to those shown by screening data



NAS Alameda, CA: UVF

UVF Screening: tPAH

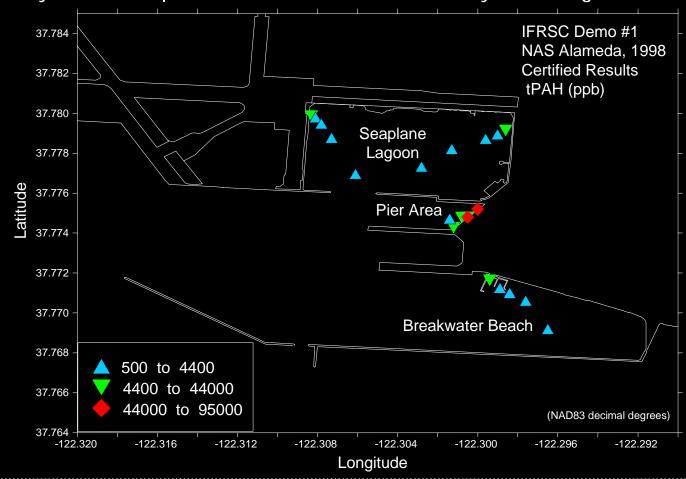
PAHs associated with pier areas and corners of lagoon



NAS Alameda, CA: UVF

Certified Results: tPAH

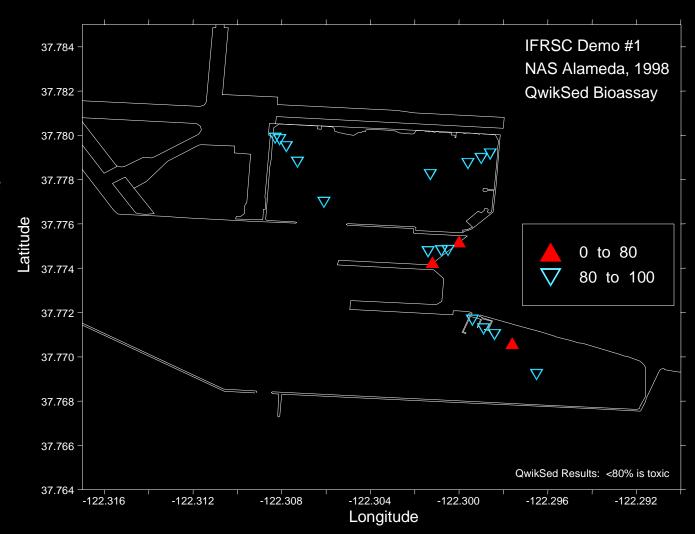
Laboratory data show patterns similar to those shown by screening data



NAS Alameda, CA: QuikSed

OwikSed Screening: Toxicity

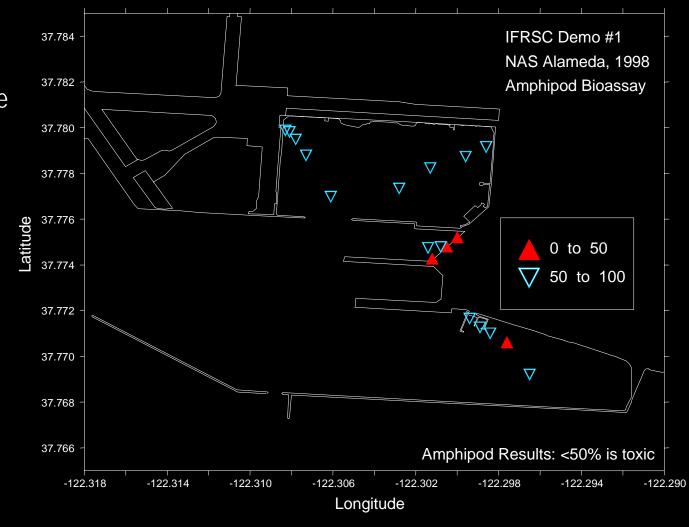
Toxicity associated with pier areas; single hits elsewhere



NAS Alameda, CA: QuikSed

Certified Results

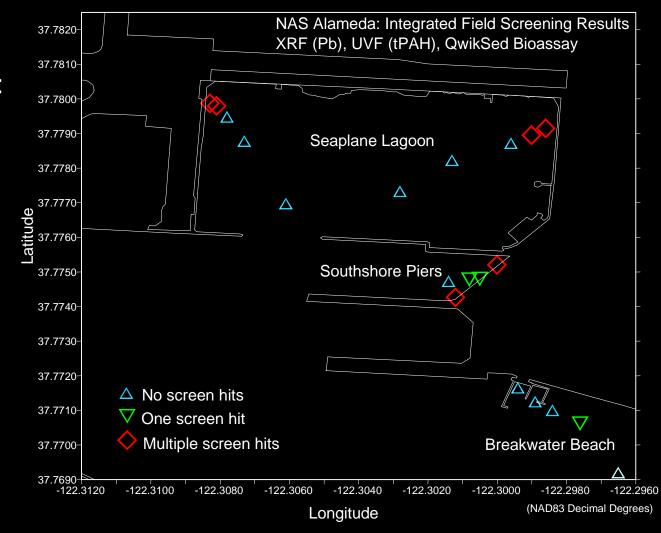
Laboratory data show patterns similar to those shown by screening data, except no toxicity in lagoon



NAS Alameda, CA: Integrated Results

Screening Criteria:

- "Hits" are defined as:
 - > ambient for Pb
 - > ambient for tPAH
 - < 80% of control for the 25% elutriate for QwikSed
- Other criteria will change contours



Integrated Field Screening Results

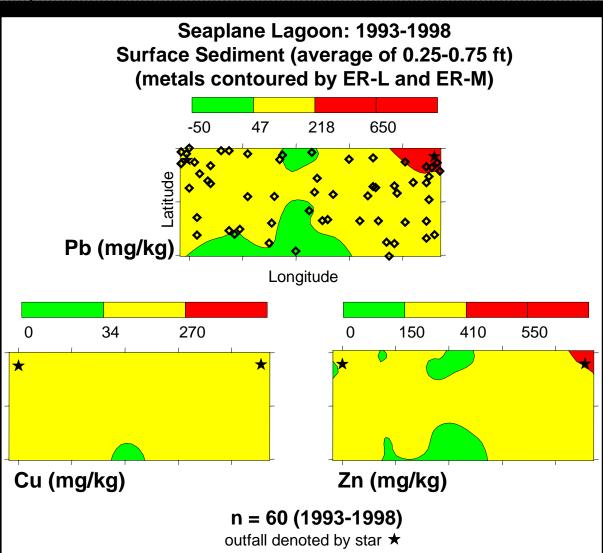
Comparisons to Standard Regulatory Data

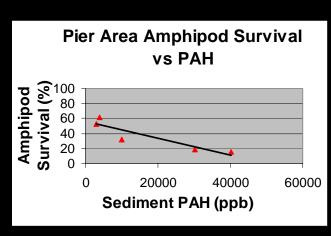
Regulatory Project:

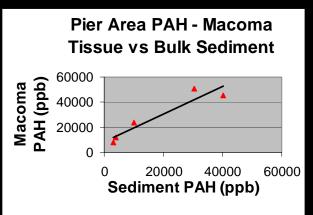
- Areas of Concern were identified in both north corners by:
 - Multiple Chemicals
 - Bioassay
 - Tissue Bioaccumulation
- Screening Results indicate the same Areas of Concern as Regulatory Project

Integrated Field Screening Results

Comparisons to Standard Regulatory Data







Outline

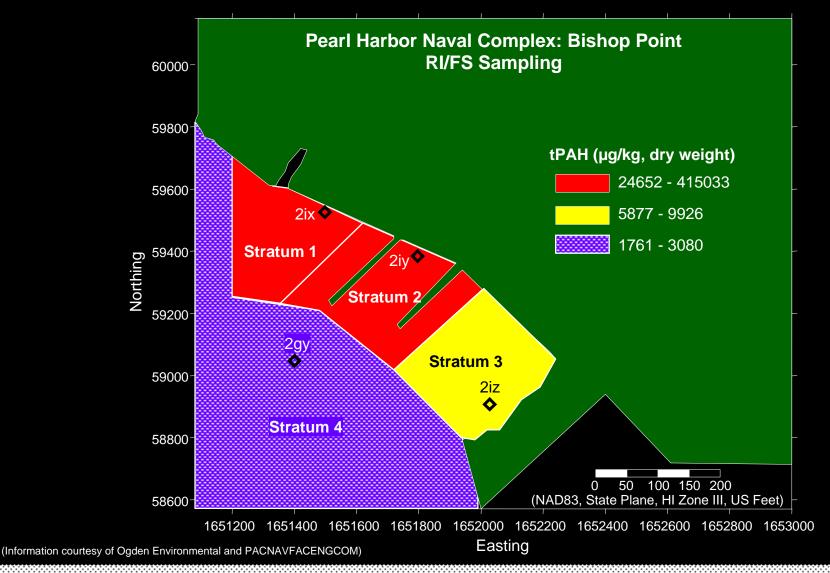
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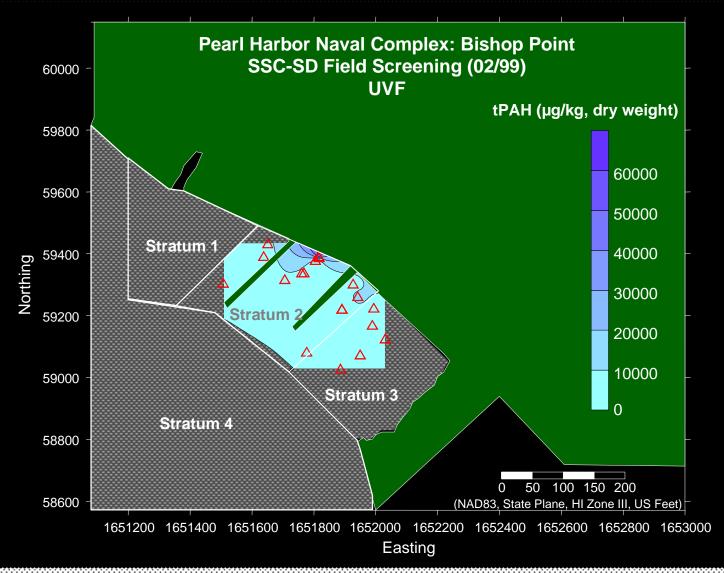
Pearl Harbor Naval Complex, HI — Bishop Point — UVF: tPAH

- Field screening for PAHs at Bishop Point can help fine-tune area of concern
- Elevated tPAH concentrations in Strata 2 and 3 appear to be associated with the inboard region (near quay wall and piers)
- This can focus and minimize the area requiring more extensive study or management

Pearl Harbor Naval Complex, HI — Bishop Point — RI/FS: tPAH



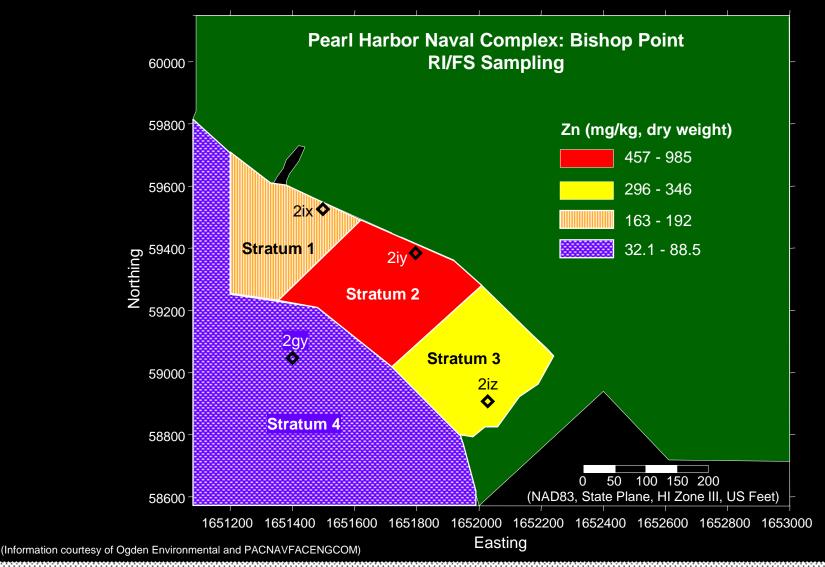
Pearl Harbor Naval Complex, HI — Bishop Point — UVF: tPAH



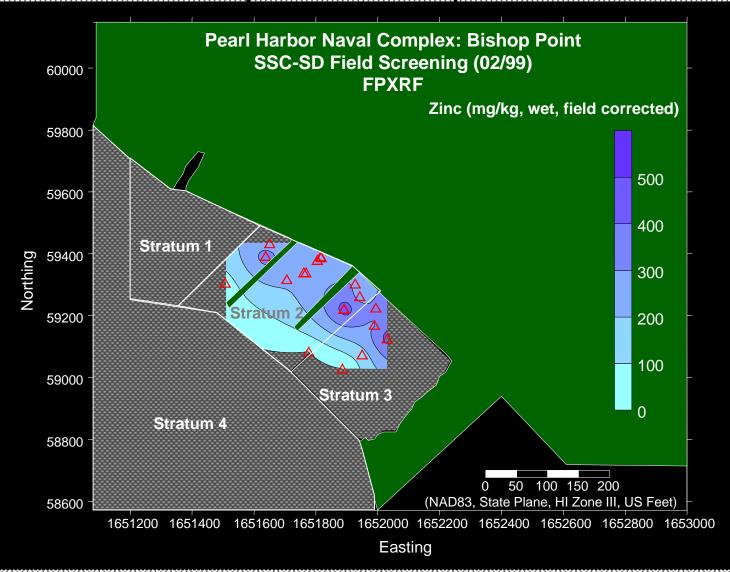
Pearl Harbor Naval Complex, HI — Bishop Point — XRF: Zinc

- Field screening for zinc at Bishop Point can help narrow region of concern
- Elevated zinc concentrations in Strata 2 and 3 appear to be associated with the inboard region (possibly associated with ships)
- Profiles suggest different sources for Zn and PAHs

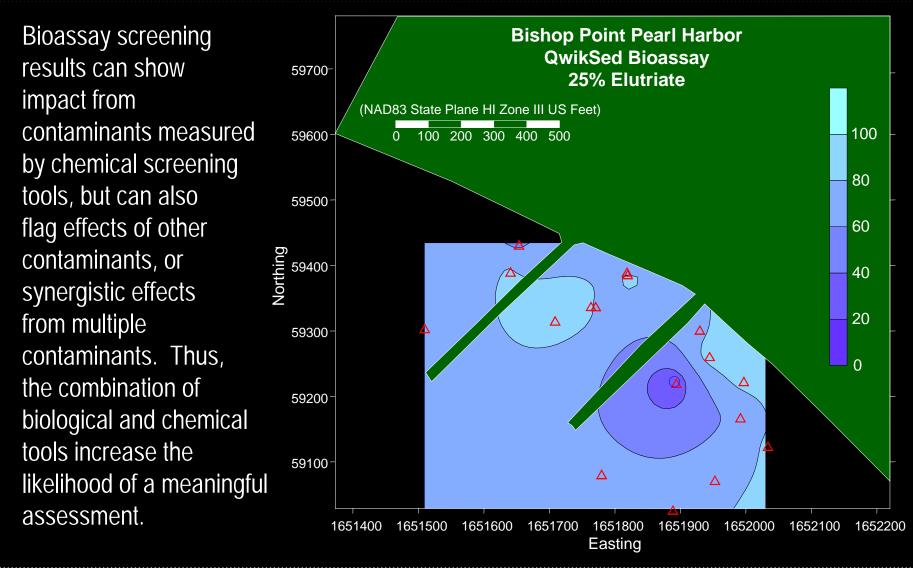
Pearl Harbor Naval Complex, HI — Bishop Point — RI/FS: Zinc



Pearl Harbor Naval Complex, HI — Bishop Point — XRF: Zinc



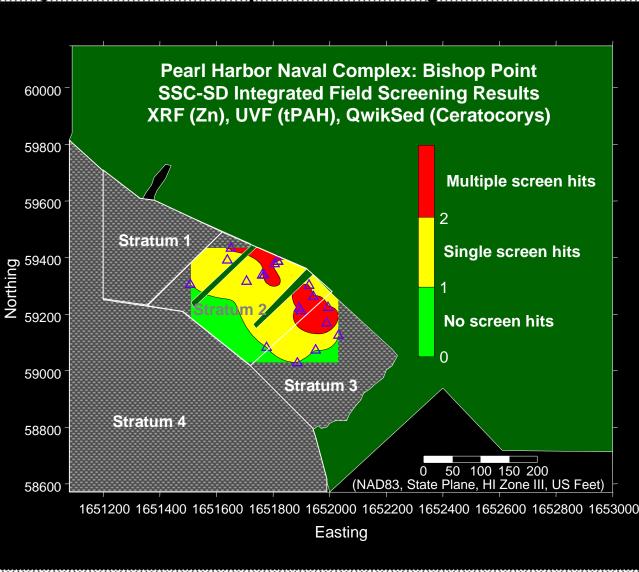
Pearl Harbor Naval Complex, HI — Bishop Point — QuikSed: Toxicity



Pearl Harbor Naval Complex, HI — Bishop Point — Integrated Results

Screening Criteria:

- "Hits" are defined as:
 - > 250 ppm for Zn
 - > ER-L for tPAH
 - < 80% of control for the 25% elutriate for QwikSed
- Other criteria will change contours



Value Added: Additional Field Screening Tools

Pearl Harbor Naval Complex, HI

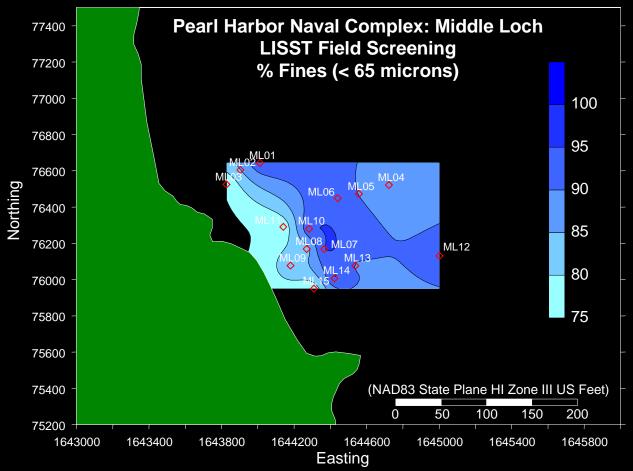
- Additional sediment field screening measurements provided better insight into site
 - Grain Size
 - Moisture Content
 - Total Ammonia
- These field screening tools brought to the site lend insight into sediment biogeochemical characteristics which can control contaminant mobility, fate, and behavior
- This additional information is important in interpreting data from the three main screening tools

Value Added: Grain Size

Pearl Harbor Naval Complex, HI

Additional Sediment Field Screening Measurements Provided Better Insight into Site

LISST: Grain Size (% Fines)

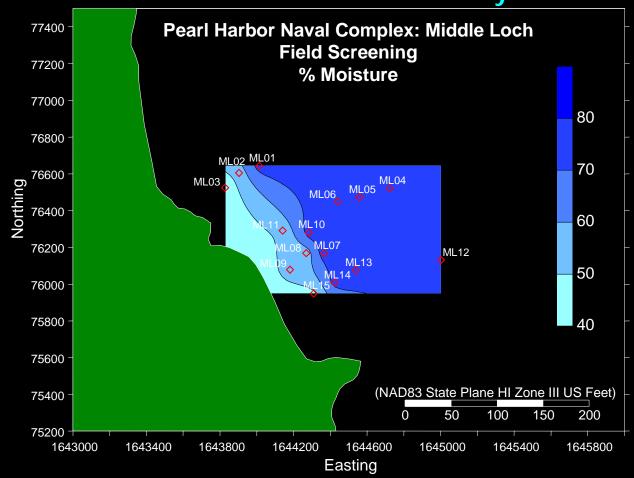


Value Added: % Moisture

Pearl Harbor Naval Complex, HI

Additional Sediment Field Screening Measurements Provided Better Insight into Site

% Moisture: IR Moisture Analyzer

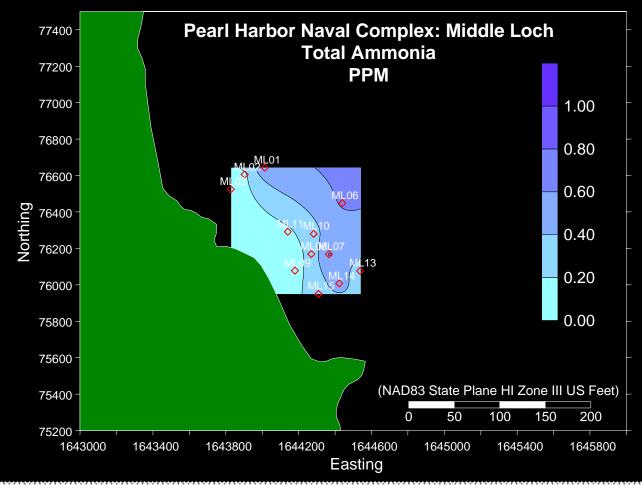


Value Added: Total Ammonia

Pearl Harbor Naval Complex, HI

Additional Sediment Field Screening Measurements Provided Better Insight into Site

Total Ammonia: Probe



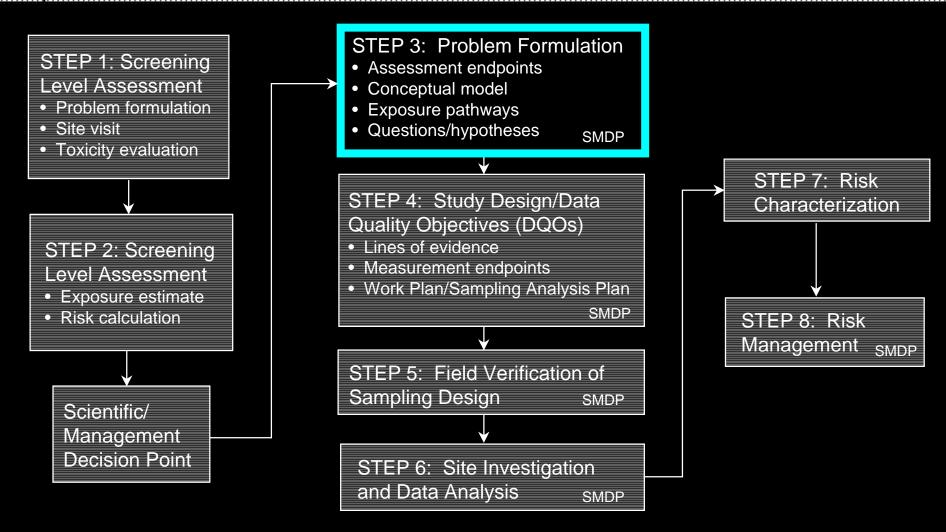
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Navy Ecological Risk Assessment Tiered Approach*

Role in the Ecological Risk Assessment Process



*CNO Letter 5090 N453E/9U595355, 05 April, 1999

Refinement of Exposure Assumptions

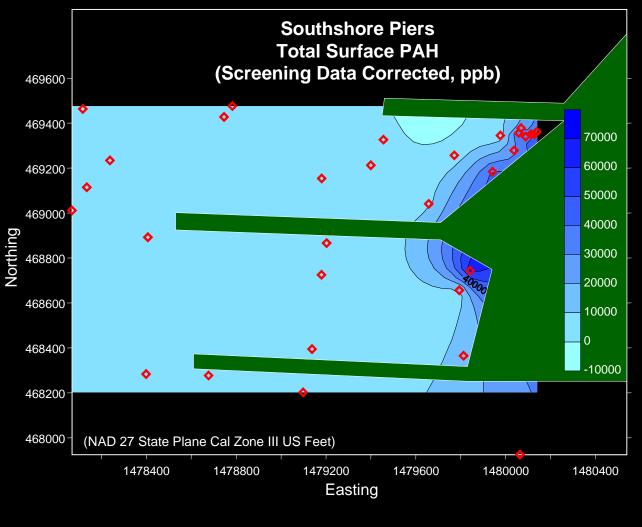
Step 3a in the Ecological Risk Assessment Process

- Refinement may include
 - Considerations of background, sample detection frequency, source, bioavailability and realistic exposure scenarios
- By generating high density contours of contaminants or toxicity
 - Random hits are de-emphasized
 - Unique sources are flagged
 - Background levels or trends are characterized
 - All this information focuses risk questions

Example: Southshore Piers

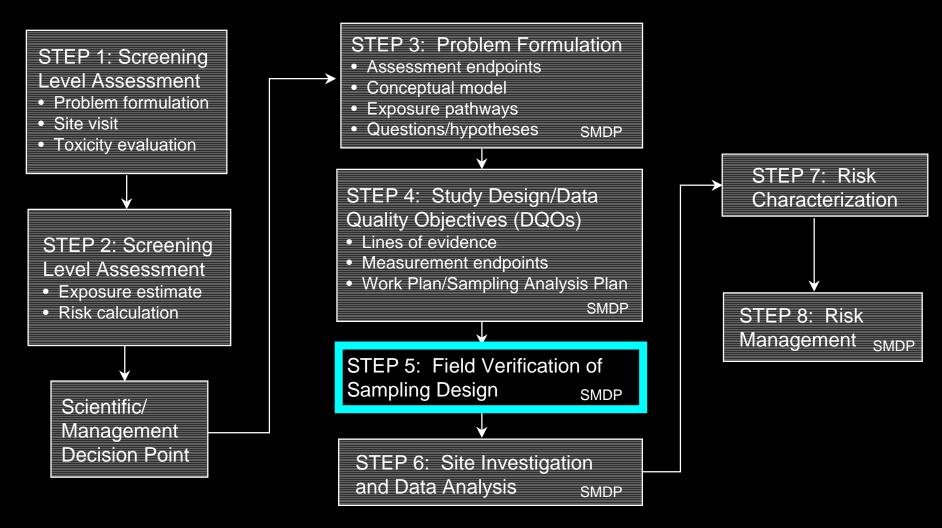
Step 3a in the Ecological Risk Assessment Process

- High PAHs at quay wall drove discussions
- One day of higher density rapid screening analysis suggested that PAHs in sediments were confined along quay wall
- Selected samples underwent laboratory analysis, which confirmed creosote impact



Navy Ecological Risk Assessment Tiered Approach*

Role in the Ecological Risk Assessment Process



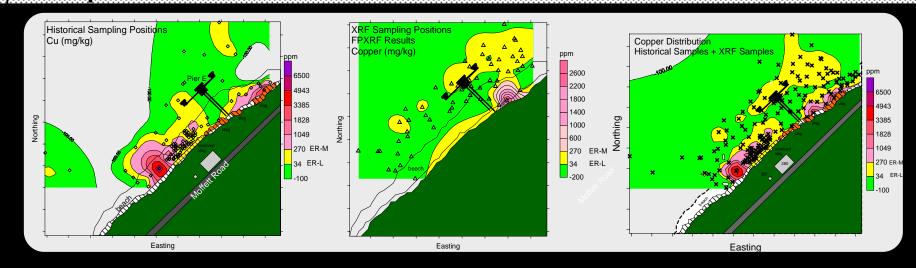
*CNO Letter 5090 N453E/9U595355, 05 April, 1999

Step 5: Verification of Field Sampling Design

Role in the Ecological Risk Assessment Process

- At times uncertainty remains on the validity of the sampling design
- Rapid characterization allows a rapid, low cost verification of the sampling design before the site investigation (sampling) and analysis (Step 6)

Example #1: North Island



Historical Data + XRF Data = Verification

- RPMs and regulators were concerned that sampling plan designed from historic data would not accurately delineate extent of contamination
- XRF was used to provide low cost, high-density data to verify sampling design

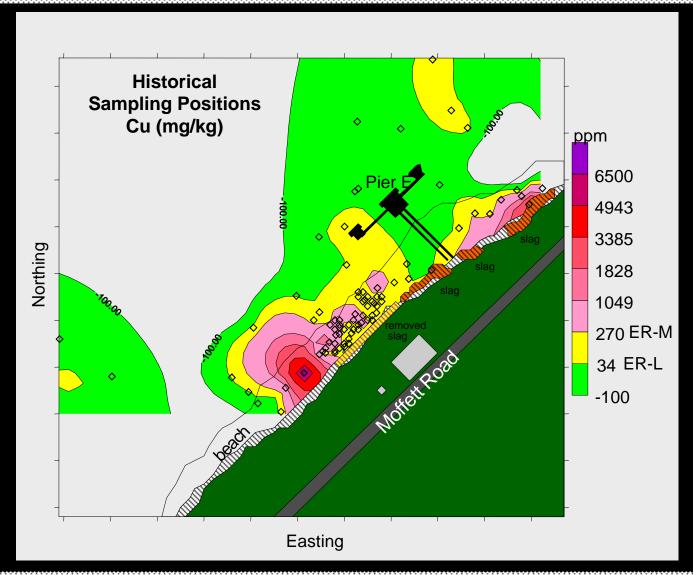
Example #1: North Island

Historical Data

+

XRF Data

Verification



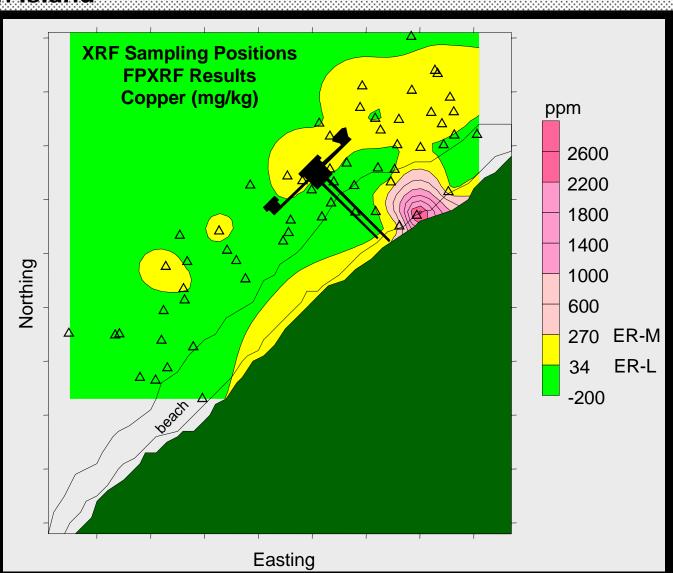
Example #1: North Island

Historical Data

+

XRF Data

Verification



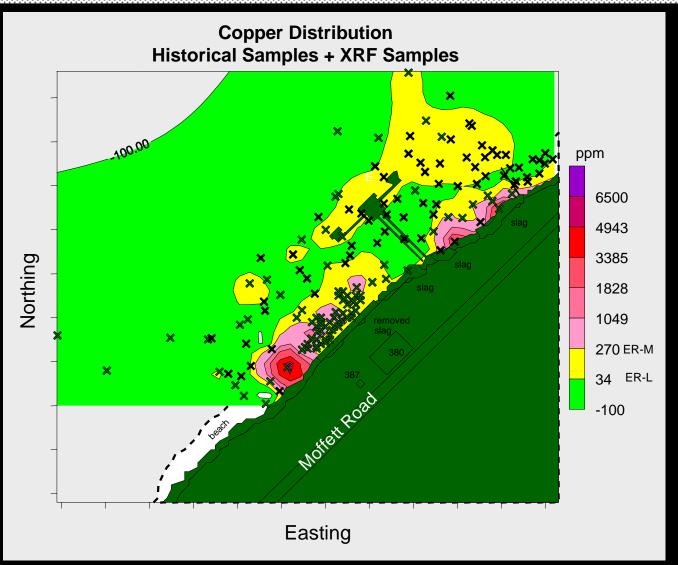
Example #1: North Island

Historical Data

+

XRF Data

Verification



Example #1: North Island

- At times, a more complete understanding of distribution and variability of sediment chemistry obtained during ERA is required in order to support sampling design established for FS work
- Rapid characterization tools can be used to provide data density necessary to support sediment contaminant mapping in a cost- and time-effective manner

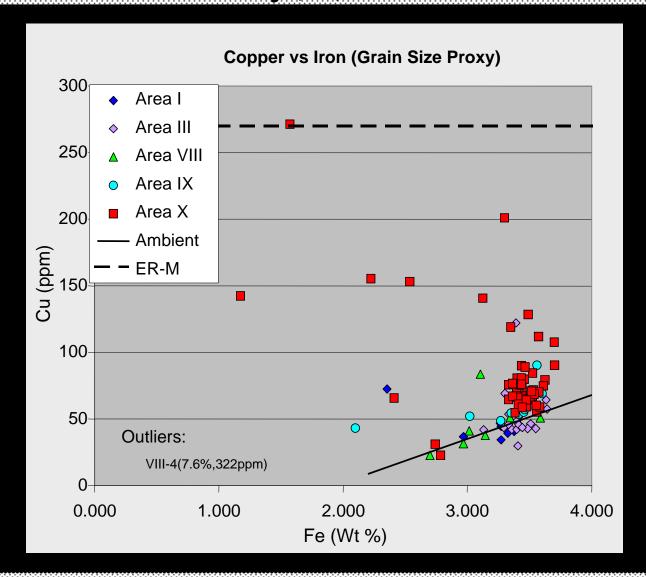
Example #2: Hunters Point Sediment Study

- Two RSC* tools (Bench-top EDXRF and Immunoassay) were used at Hunters Point Shipyard to:
 - Increase data density
 - Provide supporting data for a sediment study sample design
 - Confirm the conceptual model for the site chemistry

^{*}RSC analyses performed in laboratory, samples shipped overnight

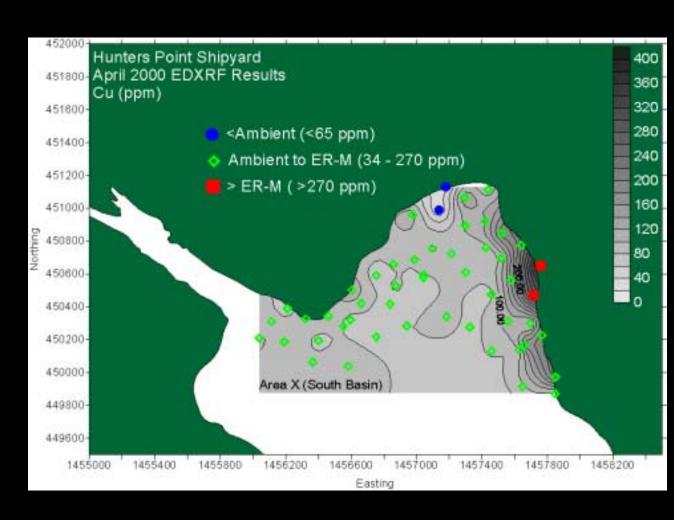
Example #2: Hunters Point Sediment Study (Cu)

Background analysis
 (Fe: Cu normalization)
 shows ambient trend
 plus some elevated
 Area X samples
 suggesting additional
 Cu sources to
 sediments



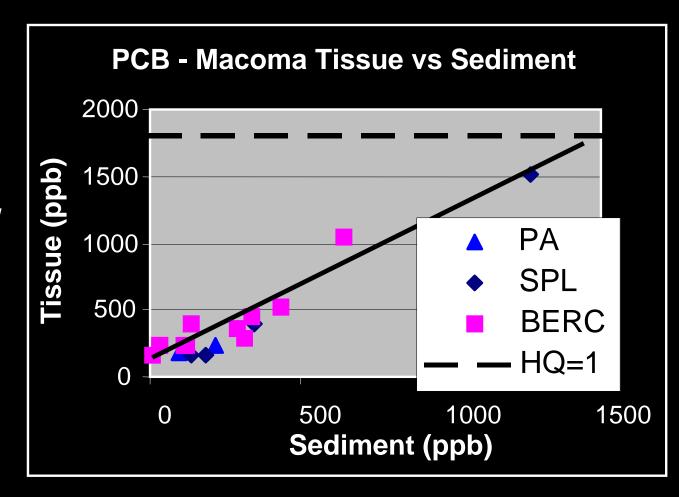
Example #2: Hunters Point Sediment Study (Cu)

Copper results compared to background and general toxicity benchmarks (ER-M from Long et al, 1995) may divide area into strata targets for standard regulatory tests



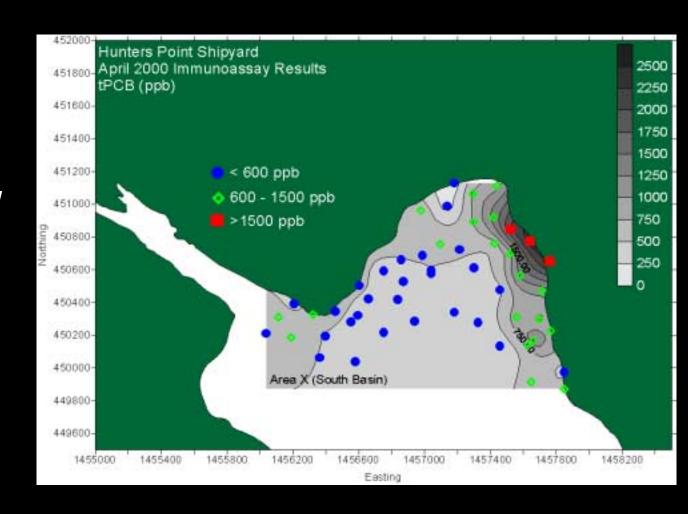
Example #2: Hunters Point Sediment Study (PCBs)

 Site-specific bioaccumulation benchmark (prey tissue level resulting in dose derived HQ=1) may indicate sediment levels of potential concern



Example #2: Hunters Point Sediment Study (PCBs)

PCB results
 compared to
 site-specific
 benchmarks may
 divide area into
 strata targets for
 standard
 regulatory test



Example #2: Hunters Point Sediment Study Results

- RSC tools used to screen 100 sediment samples (sampled + mapped results in two weeks)
- RSC data combined with historical data revealed extent of contamination
- Confirmed basic site conceptual model
- Data used to develop stratified sampling approach for regulatory project

Outline

- Navy Need
- Sediment Screening Technologies
 - X-Ray Fluorescence (XRF)
 - Ultraviolet Fluorescence (UVF)
 - Immunoassay for Organics (IAO)
 - QuikSed Biological Screen (QwikSed)
- Regulatory Issues
- Case Studies
- Role in the Ecological Risk Assessment Process
- Summary and Conclusions
- References
- Information Sources and Tech Transfer Tools

Summary and Conclusions

- RSC tools can be a useful part of a well planned, cost effective site assessment when dealing with sediments
- Direct comparison of screening data with standard laboratory data from regulatory programs and joint Navy-ESTCP programs have demonstrated where RSC data enhance traditional approaches
- Ongoing development of RSC tools/applications is acceptable (and encouraged) by regulators

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References

- U.S. EPA Methods
 - XRF Method 6200: http://www.epa.gov:80/epaoswer/hazwaste/test/6200.pdf
 - IAO Method 4020: http://www.epa.gov:80/epaoswer/hazwaste/test/4020.pdf
- QwikSed
 - ASTM Standard E1924: http://www.astm.org/cgi-bin/SoftCart.exe/DATABASE.CART/PAGES/E1924.htm?L+mystore+nnlf8277>
- Federal Regulatory Guidance Documents
 - Field Analytical Measurement Technologies, Applications and Selection:
 http://www.epa.gov/region09/ga/r9-gadocs.html
 - Field Analytical and Site Characterization Technologies, Summary of Applications: http://www.epa.gov/swertio1/techdrct/td0198.htm
- Field Analytical and Site CharacterizationTechnologies, Summary of Applications
 - http://www.epa.gov/swertio1/char.htm (search Publications, EPA-542-R-97-011)
- RSC Issue Paper
 - V.J. Kirtay and S.E. Apitz (2000) "Rapid Sediment Characterization (RSC) Tools for Ecological Risk Assessments", for Navy Guidance on Guiding Ecological Risk Assessments: http://web.ead.anl.gov/ecorisk/>

Outline

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RSC TechData Sheets

- Rapid Characterization of Metals in Sediment using X-Ray Fluorescence (XRF) Technology
 - NFESC TDS-2076-ENV
- Rapid Characterization of PAHs in Sediment using Ultraviolet Fluorescence (UVF) Technology
 - NFESC TDS-2075-ENV
- Rapid Characterization of Toxicity in Sediment using QwikSed Bioassay
 - NFESC TDS-2077-ENV

RSC Points of Contact

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